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Dioxomolybdenum(VI) complexes of hydrazones of two substituted salicylaldehydes: synthesis, structures, and catalytic properties

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Dioxomolybdenum(VI) complexes of hydrazones of two substituted salicylaldehydes. Synthesis, structures, and catalytic properties

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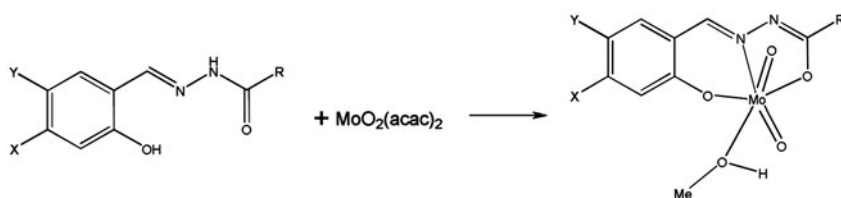
Dioxomolybdenum(VI) complexes $[\text{MoO}_2\text{L}(\text{CH}_3\text{OH})]$ ($\text{L}=\text{L}^1=N'$ -(2-hydroxy-5-nitrobenzylidene)isonicotinoylhydrazide for **1**, $\text{L}=\text{L}^2=N'$ -(4-diethylamino-2-hydroxybenzylidene)-4-methylbenzohydrazide for **2**) were prepared and structurally characterized by physicochemical and spectroscopic methods and single-crystal X-ray determination. Mo in the complexes is octahedrally coordinated. Both complexes show effective catalysis in oxidation of cyclohexene, vinylbenzene, 1-butylene, and 1-pentene, to their corresponding epoxides. In general, high epoxide yields (over 89%) and selectivity (100%) were observed for all aliphatic and aromatic substrates.

Keywords: Schiff base; Dioxomolybdenum complex; X-ray diffraction; Catalytic property

1. Introduction

Catalytic oxidation of hydrocarbons has attracted tremendous interest in catalytic and organic chemistry [1–3]. The coordination chemistry of molybdenum has special importance due to its catalytic properties in several industrial processes such as amoxidation of propene [4], epoxidation of olefins [5], olefin metathesis [6], and isomerization of allylic alcohols [7]. The first report of a molybdenum complex of multidentate ONO donor system appeared in 1990 [8]. Since then quite a number of such complexes have been reported. Schiff bases are widely used in coordination chemistry. The synthesis, characterization, and reactivity of a number of dioxomolybdenum complexes with Schiff bases have been reported [9–12]. Some of the complexes possess oxygen atom transfer properties as they were found to oxidize thiols, hydrazine, polyketones, tertiary phosphines, olefins, etc. [13–16]. As an extension of work on such fields, and to explore more efficient oxidization catalysts, in the present article, a pair of dioxomolybdenum(VI) complexes, $[\text{MoO}_2\text{L}^1(\text{CH}_3\text{OH})]$ (**1**) and $[\text{MoO}_2\text{L}^2(\text{CH}_3\text{OH})]$ (**2**) ($\text{L}^1=N'$ -(2-hydroxy-5-nitrobenzylidene)isonicotinoylhydrazide, $\text{L}^2=N'$ -(4-diethylamino-2-hydroxybenzylidene)-4-methylbenzohydrazide), have been prepared (scheme 1), characterized, and investigated for their catalytic properties.

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Scheme 1. The preparation of the complexes. For **1**: X=H, Y=NO₂, R=4-pyridine; for **2**: X=NEt₂, Y=H, R=4-methylbenzene.

2. Experimental

2.1. Materials and physical measurements

All chemical reagents were of analytical reagent grade and purchased from Xiya Chemical Reagent Company. Microanalyses (C, H, N) were performed using a Perkin-Elmer 2400 elemental analyzer. Infrared spectra were carried out using a JASCO FT-IR model 420 spectrophotometer with KBr disks from 4000 to 200 cm⁻¹. GC analyses were carried out using a Shimadzu GC-2014C gas chromatograph. ¹H NMR spectra were recorded on a Bruker instrument at 400 MHz.

2.2. Synthesis of H₂L¹ and H₂L²

H₂L¹ and H₂L² were prepared by the same method as follows. A hot methanol solution (30 mL) of substituted salicylaldehyde (10 mM) was added to a hot methanol solution (30 mL) of hydrazine derivative (10 mM). The mixtures were stirred for 30 min at reflux, and solvents were evaporated to dryness. Recrystallization from methanol gave crystalline products. Anal. Calcd for C₁₃H₁₀N₄O₄ (H₂L¹): C, 54.6; H, 3.5; N, 19.6. Found: C, 54.4; H, 3.6; N, 19.5%. ¹H NMR (DMSO-d₆): δ: 7.27 (d, 1H), 7.83 (d, 2H), 8.03 (d, 1H), 8.32 (s, 1H), 8.81 (s, 1H), 8.93 (d, 2H), 11.51 (s, 1H), 12.27 (s, 1H). Anal. Calcd for C₁₉H₂₃N₃O₂ (H₂L²): C, 70.1; H, 7.1; N, 12.9. Found: C, 69.8; H, 7.1; N, 12.8%. ¹H NMR (DMSO-d₆): δ: 1.13 (t, 6H), 2.36 (s, 3H), 3.39 (t, 4H), 6.28–6.38 (m, 2H), 7.39–7.55 (m, 3H), 7.87 (d, 2H), 8.78 (s, 1H), 11.23 (s, 1H), 12.11 (s, 1H).

2.3. Synthesis of the complexes

The complexes were prepared by the same method as follows. A hot methanol solution (15 mL) of MoO₂(acac)₂ (0.33 g, 1 mM) was added to a hot methanol solution (15 mL) of the Schiff base (1 mM). The mixtures were stirred for 30 min at reflux and then cooled to room temperature to give brown solution. Single crystals of the complexes, suitable for X-ray diffraction, were formed by slow evaporation of the methanol solutions containing the complexes in air for a few days. For **1**: Yield: 63%. Anal. Calcd for C₁₄H₁₂MoN₄O₇: C, 37.9; H, 2.7; N, 12.6. Found: C, 38.0; H, 2.8; N, 12.6%. ¹H NMR (DMSO-d₆): δ: 3.28 (s, 3H), 7.28 (d, 1H), 7.83 (d, 2H), 8.01 (d, 1H), 8.33 (s, 1H), 8.15 (s, 1H), 8.96 (d, 2H). For **2**: Yield: 45%. Anal. Calcd for C₂₀H₂₅MoN₃O₅: C, 49.7; H, 5.2; N, 8.7. Found: C, 49.6; H, 5.3; N, 8.5%. ¹H NMR (DMSO-d₆): δ: 1.13 (t, 6H), 2.36 (s, 3H), 3.27 (s, 3H), 3.38 (t, 4H), 6.25–6.41 (m, 2H), 7.36–7.59 (m, 3H), 7.85 (d, 2H), 8.23 (s, 1H).

2.4. X-ray diffraction

X-ray diffraction was performed using a Bruker APEX2 CCD diffractometer with graphite monochromated Mo K α radiation ($\lambda=0.71073$ Å) using the ω -scan technique. Determination of the Laue class, orientation matrix, and cell dimensions was performed according to established procedures where Lorentz polarization and absorption corrections were applied. Absorption corrections were applied by fitting a pseudoellipsoid to the ψ -scan data of selected strong reflections over a wide range of 2θ angles. The positions of non-hydrogen atoms were located with direct methods. Subsequent Fourier syntheses were used to locate the remaining non-hydrogen atoms. All non-hydrogen atoms were refined anisotropically. The hydroxyl H of the methanol ligands in the complexes was located from difference Fourier maps and refined isotropically, with O–H distances restrained to 0.85(1) Å and with U_{iso} fixed at 0.08 Å². The remaining hydrogens were placed in calculated positions and constrained to ride on their parent. The analysis was performed with the aid of the SHELXS-97 and SHELXL-97 suite of codes [17, 18]. Crystallographic data for the complexes are summarized in table 1. Selected bond lengths and angles are given in table 2.

2.5. Catalytic epoxidation

To a solution of olefins (0.28 mM), NaHCO₃ (0.11 mM), and catalyst (9.4×10^{-4} mM) in MeCN (0.5 mL) was added H₂O₂ (1.1 mM, 30% H₂O solution) as oxidant. After the

Table 1. Crystal data for **1** and **2**.

	1	2
Chemical formula	C ₁₄ H ₁₂ MoN ₄ O ₇	C ₂₀ H ₂₅ MoN ₃ O ₅
Fw	444.2	483.4
Crystal shape/color	Block/brown	Block/brown
Crystal size (mm)	0.30 × 0.27 × 0.27	0.20 × 0.18 × 0.17
<i>T</i> (K)	298(2)	298(2)
λ (MoK α) (Å)	0.71073	0.71073
Crystal system	Triclinic	Triclinic
Space group	<i>P</i> -1	<i>P</i> -1
<i>a</i> (Å)	6.623(2)	7.376(1)
<i>b</i> (Å)	10.732(2)	9.932(1)
<i>c</i> (Å)	12.895(3)	14.889(2)
α (°)	69.684(2)	102.013(2)
β (°)	86.642(2)	100.958(2)
γ (°)	76.170(2)	106.184(1)
<i>V</i> (Å ³)	834.4(3)	988.2(2)
<i>Z</i>	2	2
μ (MoK α) (cm ⁻¹)	0.833	0.702
<i>T</i> (min)	0.7882	0.8724
<i>T</i> (max)	0.8064	0.8900
<i>D</i> _c (g cm ⁻³)	1.768	1.624
Reflections/parameters	3545/239	4174/269
Restraints	1	1
Unique reflections	3000	3504
Goodness of fit on <i>F</i> ²	1.066	1.062
<i>R</i> _{int}	0.0226	0.0192
<i>R</i> ₁ [<i>I</i> ≥ 2σ(<i>I</i>)]	0.0332	0.0476
<i>wR</i> ₂ [<i>I</i> ≥ 2σ(<i>I</i>)]	0.0700	0.1226
<i>R</i> ₁ (all data)	0.0423	0.0576
<i>wR</i> ₂ (all data)	0.0736	0.1306

Table 2. Selected bond lengths (Å) and angles (°) for **1** and **2**.

1			
Bond lengths			
Mo1–O3	1.9424(19)	Mo1–O4	2.0095(19)
Mo1–O5	1.692(2)	Mo1–O6	2.316(2)
Mo1–O7	1.691(2)	Mo1–N1	2.242(2)
Bond angles			
O7–Mo1–O5	105.48(11)	O7–Mo1–O3	103.21(9)
O5–Mo1–O3	97.85(10)	O7–Mo1–O4	97.94(9)
O5–Mo1–O4	97.75(10)	O3–Mo1–O4	149.22(8)
O7–Mo1–N1	162.67(10)	O5–Mo1–N1	90.16(9)
O3–Mo1–N1	81.50(8)	O4–Mo1–N1	72.08(8)
O7–Mo1–O6	86.57(10)	O5–Mo1–O6	167.84(9)
O3–Mo1–O6	80.58(8)	O4–Mo1–O6	78.62(8)
N1–Mo1–O6	77.68(8)		
2			
Bond lengths			
Mo1–O1	2.013(3)	Mo1–O2	1.924(3)
Mo1–O3	1.685(3)	Mo1–O4	1.712(3)
Mo1–O5	2.365(3)	Mo1–N2	2.201(3)
Bond angles			
O3–Mo1–O4	105.18(16)	O3–Mo1–O2	98.41(15)
O4–Mo1–O2	104.70(14)	O3–Mo1–O1	97.25(14)
O4–Mo1–O1	95.18(13)	O2–Mo1–O1	150.38(13)
O3–Mo1–N2	96.94(14)	O4–Mo1–N2	155.60(14)
O2–Mo1–N2	81.68(12)	O1–Mo1–N2	71.57(12)
O3–Mo1–O5	173.45(13)	O4–Mo1–O5	80.85(13)
O2–Mo1–O5	82.27(12)	O1–Mo1–O5	79.44(11)
N2–Mo1–O5	76.68(11)		

reaction was over, for the products analysis, the solution was subjected to multiple ether extraction and the extract was concentrated to 0.5 mL by distillation in a rotary evaporator at room temperature, and then, a sample (2 μ L) was taken from the solution and analyzed by GC. The retention times of the peaks were compared with those of commercial standards and chlorobenzene was used as an internal standard for GC yield calculation.

3. Results and discussion

Schiff bases H_2L^1 and H_2L^2 were readily prepared by condensation of equimolar quantities of 5-nitrosalicylaldehyde with isonicotinohydrazide, and 4-diethylaminosalicylaldehyde with 4-methylbenzohydrazide, respectively, in methanol. The complexes were synthesized by reactions of $MoO_2(acac)_2$ with H_2L^1 and H_2L^2 , respectively, in methanol in a 1 : 1 M ratio at reflux. Chemical formulas of the complexes have been confirmed by elemental analyses, IR spectra, and X-ray single-crystal structure determination.

3.1. Structure description of the complexes

Molecular structures of **1** and **2** are presented in figures 1 and 2, respectively. The coordination geometry around each Mo can be described as slightly distorted octahedral, with one phenolate O, one imino N, and one enolate O of the dianionic Schiff-base ligand,

and with one oxo O defining the equatorial plane, and with methanol and the other oxo occupying axial positions. The Schiff-base ligands coordinate to Mo atoms in a meridional fashion forming five- and six-membered chelate rings with bite angles of 72.1(1) and 81.5(1)° for **1** and 71.6(1) and 81.7(1)° for **2**, which are similar to those observed in other molybdenum complexes [19, 20]. The displacement of Mo atoms from the equatorial mean plane toward the axial oxo is 0.290(2) Å for **1** and 0.330(2) Å for **2**. The Schiff-base ligands are coordinated in their dianionic enolate forms, which are evident from the bond lengths of C8–O4 (1.317(3) Å) and C8–N2 (1.295(3) Å) for **1** and C8–O1 (1.322(5) Å) and C8–N1 (1.297(5) Å) for **2**. The C7–N1 bond in **1** is shorter than the C9–N2 bond in **2**, which is influenced by the electron withdrawing NO₂ in **1** and electron donating NEt₂ in **2**. For the Schiff base of **1**, the dihedral angle between the benzene ring and the pyridine ring is 8.5(3)°. For the Schiff base of **2**, the dihedral angle between the two benzene rings is 5.4(3)°. The Mo–O, Mo–N, and Mo=O bonds in both complexes are within normal ranges and comparable to each other and are similar to those observed in dioxomolybdenum(VI) complexes with Schiff bases [21–23].

In the crystal structure of **1**, two adjacent molecules are linked through intermolecular O6–H6···N3ⁱ hydrogen bonds [O6–H6=0.85(1) Å, H6···N3ⁱ=1.859(11) Å, O6···N3ⁱ=2.698(3) Å, O6–H6···N3ⁱ=175(4)°; symmetry code for i: 2–x, –y, 2–z] to form a dimer, as shown in figure 3. In the crystal structure of **2**, two adjacent molecules are linked through intermolecular O5–H5···N1ⁱⁱ hydrogen bonds [O5–H5=0.85(1) Å, H5···N1ⁱⁱ=1.918(16) Å, O5···N1ⁱⁱ=2.754(4) Å, O5–H5···N1ⁱⁱ=170(6)°; symmetry code for i: 1–x, –y, –z] to form a dimer, as shown in figure 4.

3.2. Infrared spectra

Medium and broad bands centered at 3434 cm⁻¹ (**1**) and 3326 cm⁻¹ (**2**) in IR spectra of the complexes can be assigned to ν_{OH} of methanol. The Mo=O stretching modes occur as a pair of sharp strong bands at 936 and 921 cm⁻¹ for **1** and at 934 and 890 cm⁻¹ for **2**, assigned to the anti-symmetric and symmetric stretching modes of the dioxomolybdenum (VI) [24]. The difference between the two peaks in **2** is bigger than that in **1**, in accord with the Mo=O bond lengths, much more different in **2** than those in **1**. The intense bands indicative of –C=N– are located at 1605 cm⁻¹ for **1** and 1613 cm⁻¹ for **2**, much lower

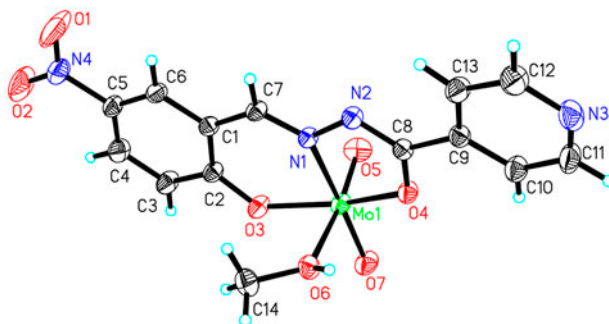


Figure 1. Molecular structure of **1** with 30% probability thermal ellipsoids.

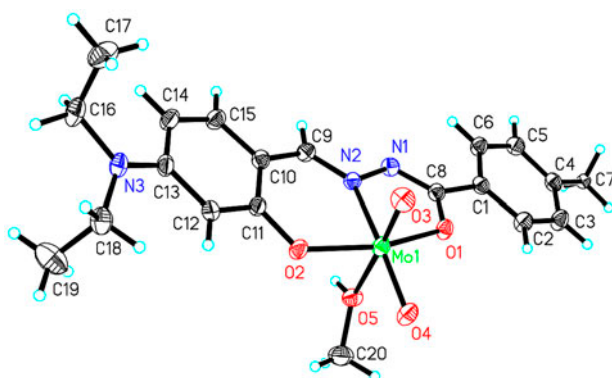


Figure 2. Molecular structure of **2** with 30% probability thermal ellipsoids.

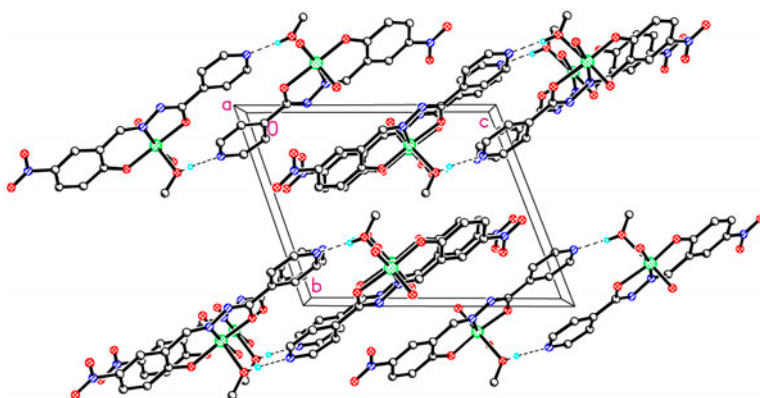


Figure 3. Molecular packing of **1**. Hydrogen bonds are drawn as thin dashed lines.

than those of the free Schiff base. New weak peaks observed at $600\text{--}400\text{ cm}^{-1}$ may be attributed to the Mo–O and Mo–N vibrations in the complexes.

3.3. Catalytic results

Both complexes show effective catalysis in the oxidation of cyclohexene, vinylbenzene, 1-butylene, and 1-pentene, to their corresponding epoxides. The detailed information of catalysis is given in table 3. In general, high epoxide yields and selectivity were observed for all aliphatic and aromatic substrates. The catalytic studies using the complexes revealed that the efficiency of catalysts toward all substrates are attractive with high conversion, TON, and selectivity. In general, **1** has better catalytic properties than **2**. This might be caused by the electron withdrawing NO_2 in **1**, which made HCO_4^- more readily coordinate to Mo. Despite having different substituents of the hydrazones, both complexes show effective catalytic properties. The complexes in the present work have similar catalytic properties to those of a similar molybdenum(VI) catalyst without any substituent in the hydrazone ligand [20] and of a multi-wall carbon nanotube supported tungsten

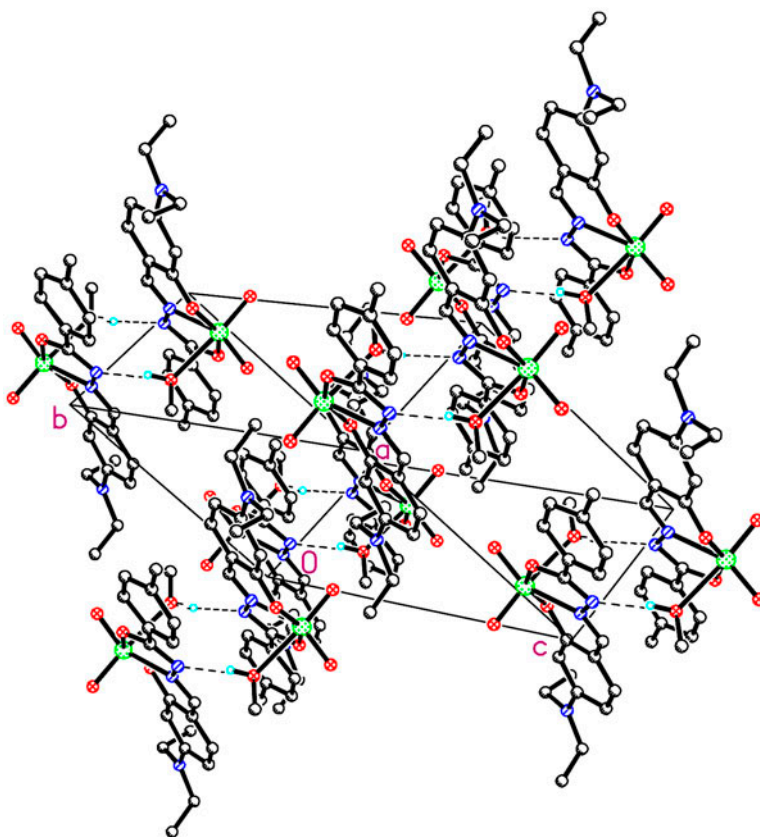




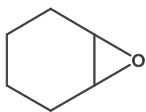
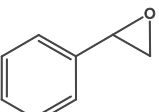
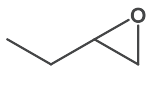
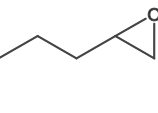


Figure 4. Molecular packing of 2. Hydrogen bonds are drawn as thin dashed lines.

Table 3. Detailed information of the catalytic oxidation of olefins catalyzed by the complexes.^a

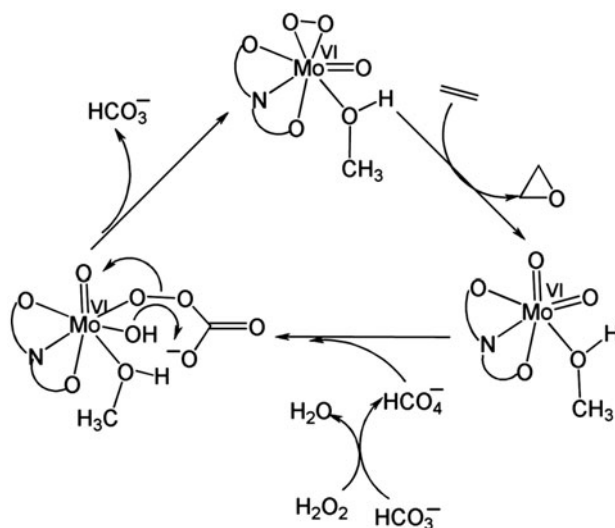
Substrate					
Product					
Conversion (%) ^b	1	93 (267)	97 (283)	95 (307)	98 (297)
(TON) ^c	2	89 (253)	90 (290)	90 (298)	93 (282)
Selectivity (%) ^d	1	100	100	100	100
	2	100	100	100	100

^aThe molar ratios for catalyst : substrate : NaHCO₃ : H₂O₂ are 1 : 298 : 117 : 1170. The reactions were performed in (70 : 30) mixture of CH₃OH/CH₂Cl₂ (1.2 mL) under air at room temperature.

^bThe GC conversion (%) is measured relative to the starting olefin after 75 min.

^cTON = (mM of product)/mM of catalyst.

^dSelectivity to epoxide = (epoxide% / (epoxide% + aldehyde%)) × 100.



Scheme 2. Possible catalytic mechanism of olefins to epoxides.

hexacarbonyl [25]. The complexes have higher conversion values than those of vanadium (IV) complexes with Schiff bases [26] and multi-wall carbon nanotube supported manganese(III) tetraphenylporphyrin [27]. The mechanism (scheme 2) for epoxidation of various olefins to epoxides using the complexes as catalysts has been proposed by comparison with the literature [28]. When H_2O_2 was used as a sole oxidant, the catalytic efficiency is not high, but when NaHCO_3 was added as a co-catalyst the efficiency of the system increases many fold. The key aspect of such a reaction is that H_2O_2 and hydrogen carbonate react in an equilibrium process to produce peroxy-monocarbonate, HCO_4^- , which is a more reactive nucleophile than H_2O_2 and speeds the epoxidation [28]. The basic principle of the catalytic reaction is the conversion of oxoperoxo-complex to dioxo-complex transferring oxo species to the olefins and conversion of dioxo-complex to the oxoperoxo-complex reacting with HCO_4^- to regain the catalytic activity.

4. Conclusion

Two new dioxomolybdenum(VI) complexes derived from Schiff bases, *N'*-(2-hydroxy-5-nitrobenzylidene)isonicotinoylhydrazide and *N'*-(4-diethylamino-2-hydroxybenzylidene)-4-methylbenzohydrazide, were prepared and structurally characterized. The Schiff-base ligands coordinate to Mo through phenolate O, imino N, and enolate O. Mo in the complexes have octahedral coordination. The complexes show effective catalysis in oxidation of several olefins to their corresponding epoxides. Further work needs to be carried out to explore more efficient catalysts based on the present results.

Supplementary materials

CCDC 909611 (1) and 909612 (2) contain the supplementary crystallographic data for the complexes. These data can be obtained free of charge via <http://www.ccdc.ac.uk/conts/retrieving.html>, or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336 033; or E-mail: deposit@ccdc.cam.ac.uk.

References

- [1] N. Mizuno, K. Kamata. *Coord. Chem. Rev.*, **255**, 2358 (2011).
- [2] S. Masoudian, H.H. Monfared. *Transition Met. Chem.*, **37**, 37 (2012).
- [3] S.I. Chan, C.Y.C. Chien, C.S.C. Yu, P. Nagababu, S. Maji, P.P.Y. Chen. *J. Catal.*, **293**, 186 (2012).
- [4] R.K. Grasselli. *Catal. Today*, **49**, 141 (1999).
- [5] R.J. Cross, P.D. Newman, R.D. Peacock, D. Stirling. *J. Mol. Catal.*, **144**, 273 (1999).
- [6] K.J. Ivin, J.C. Mol. *Olefin Metathesis Polymerization*, Academic Press, London (1997).
- [7] J. Belgacem, J. Kress, J.A. Osborn. *J. Am. Chem. Soc.*, **114**, 1501 (1992).
- [8] S.A. Roberts, C.G. Young, C.A. Kipke, W.E. Cleland, K. Yamanouchi, M.D. Carducci, J.H. Enemark. *Inorg. Chem.*, **29**, 3650 (1990).
- [9] N.K. Ngan, K.M. Lo, C.S.R. Wong. *Polyhedron*, **33**, 235 (2012).
- [10] T.A. Youssef. *J. Coord. Chem.*, **61**, 816 (2008).
- [11] B.T. Thaker, R.S. Barvalia. *J. Coord. Chem.*, **63**, 1597 (2010).
- [12] S.A. Ali, A.A. Soliman, M.M. Aboaly, R.M. Ramadan. *J. Coord. Chem.*, **55**, 1161 (2002).
- [13] N.-Y. Jin, W.-H. Li. *Synth. React. Inorg. Met.-Org. Nano-Met. Chem.*, **42**, 1167 (2012).
- [14] W.-X. Xu, W.-H. Li. *Synth. React. Inorg. Met.-Org. Nano-Met. Chem.*, **42**, 160 (2012).
- [15] J. Liimatainen, A. Lehtonen, R. Sillanpaa. *Polyhedron*, **19**, 1133 (2000).
- [16] K. Ambroziak, R. Pelech, E. Milchert, T. Dziembowska, Z. Rozwadowski. *J. Mol. Catal. A: Chem.*, **211**, 9 (2004).
- [17] G.M. Sheldrick. *SHELX-97, Program for crystal structure solution and refinement*, Göttingen University, Germany (1997).
- [18] G.M. Sheldrick. *SHELXTL (Version 5)*, Siemens Industrial Automation Inc., Madison, WI, (1995).
- [19] R. Dinda, P. Sengupta, S. Ghosh, H. Mayer-Figge, W.S. Sheldrick. *J. Chem. Soc., Dalton Trans.*, 4434 (2002).
- [20] M. Bagherzadeh, M. Amini, H. Parastar, M. Jalali-Heravi, A. Ellern, L.K. Woo. *Inorg. Chem. Commun.*, **20**, 86 (2012).
- [21] R.D. Chakravarthy, K. Suresh, V. Ramkumar, D.K. Chand. *Inorg. Chim. Acta*, **376**, 57 (2011).
- [22] A.K. Sah, C.P. Rao, P.K. Saarenketo, E.K. Wegelius, E. Kolehmainen, K. Rissanen. *Eur. J. Inorg. Chem.*, **11**, 2773 (2001).
- [23] M. Minelli, F. Namuswe, D. Jeffrey, A.L. Morrow, I.A. Guzei, D. Swenson, E. Bothe, T. Weyhermuller. *Inorg. Chem.*, **45**, 5455 (2006).
- [24] T. Glowiak, L. Jerzykiewicz, J.M. Sobczak, J.J. Ziolkowski. *Inorg. Chim. Acta*, **356**, 387 (2003).
- [25] M. Nooraiepour, M. Moghadam, S. Tangestaninejad, V. Mirkhani, I. Mohammadpoor Baltork, N. Iravani. *J. Coord. Chem.*, **65**, 226 (2012).
- [26] A. Ghaffari, M. Behzad, G. Dutkiewicz, M. Kubicki, M. Salehi. *J. Coord. Chem.*, **65**, 840 (2012).
- [27] M. Zakeri, M. Moghadam, I. Mohammadpoor-Baltork, S. Tangestaninejad, V. Mirkhani, A.R. Khosropour. *J. Coord. Chem.*, **65**, 1144 (2012).
- [28] S.K. Maiti, S. Dinda, S. Banerjee, A.K. Mukherjee, R. Bhattacharyya. *Eur. J. Inorg. Chem.*, **12**, 2038 (2008).